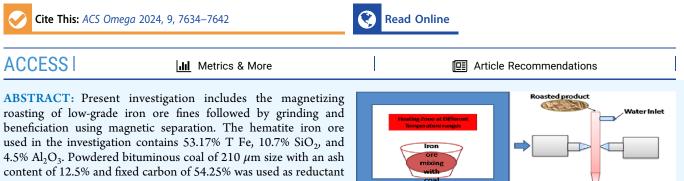


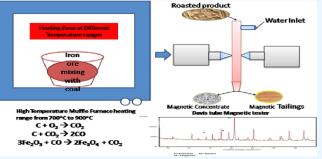
Article

Beneficiation of Low-Grade Hematite Iron Ore Fines by Magnetizing **Roasting and Magnetic Separation**

Prabhu Chand Kukkala, Shravan Kumar,* Akhileshwar Nirala, Mohammad Amir Khan, Meshel Q. Alkahtani, and Saiful Islam



during magnetizing roasting. Optical microstructures have shown where iron and silicate minerals are found and how they are interconnected. Hematite is the most abundant material in the specimen and is found in fine- and medium-sized grains. Hematite emerged as the predominant iron-bearing mineral, accompanied by magnetite and goethite phases in smaller proportions according to



XRD analyses. The primary gangue mineral identified by scanning electron microscopy is quartz, with gibbsite, feldspar, and pyrolusite present in lesser levels. The effects of iron/coal ratio, roasting time, and roasting temperature were considered as variable parameters. Hematite ore's magnetic characteristics were significantly impacted by magnetizing roasting. By selectively magnetizing roasting, hematite is transformed into magnetite. With an Fe grade of 65.25% at a recovery value of 72.5% in the concentrate, magnetic separation produced the greatest result for Fe. The performance of magnetization and therefore the magnetic separation process were shown to be significantly impacted by temperature, reductant %, and roasting duration in this investigation.

1. INTRODUCTION

The transformation of hematite into goethite is a result of weathering processes in iron ore formation. The combination of intricate, crystalline goethite with hematite is termed vitreous goethite. When goethite is found in claylike substances such as gibbsite and kaolinite, it is termed ochreous goethite. Beneficiation of low-grade iron ore fines is considered to be a challenging task due to the smaller grain size and their complex association with the gangue matrix. Iron ore of a specified grade constitutes the principal raw material for the iron and steel sector. However, due to low availability of high-grade resources, we are heading toward the beneficiation of secondary iron ore resources to get the desired quality. These resources mainly include low grade iron ores fines (-6)mm) and slimes (-150 μ m). Indian iron ore typically comprises of good grade of iron along with relatively high amount of SiO_2 and Al_2O_3 as the major tailor materials. Hematite iron ore in India is found in various forms, including banded, laminated, friable, and even powdered form. Hence, during the mining operations, crushing, screening, handling, and transportation, a significant fraction of fines is generated that is approximately 35-50% of the total iron ore mined. These ores are typically left untreated at the mining site due to

a lack of appropriate beneficiation technology presence in the mining sector.¹

Hematite constitutes the predominant iron ore material in India; however, the reserves of high-grade hematite are diminishing rapidly. Consequently, India has shifted its focus to using low-grade minerals as the primary source for iron and steel production. Our research and efforts focused on exploring these lower-quality iron ore deposits due to the scarcity of high-grade resources in the country. These subpar deposits are defined by a notable presence of goethite, kaolinite, and gibbsite.² Additionally, the consumption of low-quality iron ore, typically discarded as tailings, fines, and slimes, by industries engaged in iron ore beneficiation poses inevitable challenges, in terms of the exhaustion of high-quality iron ore reserves. Another challenge arises from the increasing demand for steel. In general, the practice adopted by major steel

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industries in India is to use 60+ Fe grade iron ore feed stocks to obtain the best productivity from blast furnaces. The diminishing availability of diminishing reserves of high-quality iron ore deposits compel steel manufacturers to turn to lowgrade iron ores to fulfill their raw material needs. However, the complex mineralogical characteristics of most low-quality iron ores, coupled with the limitations of existing technology, make their efficient utilization a formidable task.³ This in efficiency results in the generation of substantial tailings, occupying significant space, causing environmental harm, and leading to the waste of valuable iron resources. Consequently, the advancement of beneficiation technology is imperative as a strategic approach to address the aforementioned issues. Some researchers have prioritized the integration of three primary approaches, namely, flotation, magnetic separation, and gravity separation, for the separation of low-quality iron ore deposits due to advancements in standard beneficiation technologies. Roy et al.⁴ employed traditional beneficiation equipment, specifically a table concentrator, to extract low-quality iron ores, characterized by elevated hematite concentrations. Their findings underscored the significance of ore characteristics in the separation process, with the separation of ores containing a substantial goethite component proving more intricate than those with a high hematite concentration due to goethite's delicate nature.4

Chaurasia and Nikkam emphasized the utility of centrifugal force in separating low-grade fines from iron ore, as it facilitates a faster settling rate of mineral particles compared to traditional gravity methods.⁵ The implementation of a multigravity separator significantly improved the quality of low-quality iron ore powders, elevating the ore of iron grade from 50.74 to 65.11% with an impressive iron retrieval rate of 71.88%. Several techniques have been utilized to improve the quality of inferior iron ores, encompassing magnetic separation, gravity separation using hydrocyclones, spiral concentrators, and jigs, as well as flotation processes.⁶⁻¹¹ Despite achieving iron ore concentrate grades comparable to those employed in blast furnace operations, the challenges of insufficient iron recovery and significant iron wastage persisted. This issue may stem from the intricate interactions among gangue minerals and the prevalence of abundant gangue materials and fine iron particles within low-quality iron ores. Moreover, the prevalence of goethite and limonite, which are the primary iron ores in lower-quality deposits, tends to generate slimes during fine grinding, hindering effective separation. Consequently, conventional beneficiation techniques face unique challenges in effectively separating low-quality iron ores.

Numerous studies have highlighted the potential of converting iron minerals within goethite iron ores into magnetite iron through reduction processes involving coal. This approach addresses the limitations associated with conventional beneficiation techniques, such as extended processing times and suboptimal iron recovery rates when dealing with goethite iron ores.^{11,12}

Recent pilot testing conducted by Zhong demonstrated the production of hematite ore powder with an impressive overall recovery rate of 85.61% by employing coal-based reduction on goethite iron ores, followed by magnetic separation.¹³ Direct reduction experiments on goethite ores frequently yield favorable iron recovery rates while maintaining efficient process flow. Nonetheless, It is essential to emphasize that employing elevated temperatures and prolonged reaction times

can escalate energy consumption, especially when processing low-grade iron ores containing a high proportion of gangue minerals. Hence, it becomes imperative to explore high-efficiency, low-energy methods for beneficiation of low-quality ores of iron. $^{\rm 12,14}$

The beneficiation strategy adopted in India is broadly classified into three categories: -40 + 10 mm under coarse lump, -10 + 0.15 mm under fines, and -0.15 under slimes. The coarse lump is fed directly to blast furnace operations, fines with 3-5% Al₂O₃ are treated in sinter plants. However, the slimes with 6-8% Al₂O₃ that accounts 20-25% of total iron ore mined is dumped in tailing ponds as slimes.¹⁵ The available beneficiation methods for treatment of these slimes by gravity separation, magnetic separation, froth flotation, selective flocculation, etc., have limitations due to various physical properties, ore complex mineralogy, and composition. 16,17 Additionally, the presence of high $\rm Al_2O_3$ content in the slimes in the form of kaolinite and gibbsite poses massive environmental hazard.¹⁵ Researchers have investigated the beneficiation of fines by a sequential process involving gravity separation, followed by magnetic separation, and concluding with flotation and have shown the possibility to improve the grade of iron ore containing 35% Fe and 45% SiO2 to concentrate with more than 64% Fe.¹⁸ However, the fines (-1)mm +0.15 mm) and slimes (-0.15 mm) are difficult to process with conventional beneficiation methods.^{19–2}

The emerging concept in the beneficiation of low-grade iron ore fines involves magnetizing roasting followed by magnetic separation and is expected to give prominent results.^{22–27} Studies have been carried out to explain the various aspects of this process in the recent past for iron ore fines of different grades.^{28–32} However, very limited studies are available that show the use of low cost easily available coal fines in magnetizing roasting as reducing agent.

In the study, a process involving reduction roasting followed by low-intensity magnetic separation was utilized to obtain an iron concentrate suitable for use as a blast furnace feedstock. Reduction of iron oxides is a stepwise process from Fe_2O_3 to Fe as shown

$$C + O_2 \rightarrow CO_2$$

 $CO_2 + C \rightarrow 2CO$
 $3Fe_2O_3(s) + CO(g) \rightarrow 2Fe_3O_4(s) + CO_2(g)$

This paper mainly highlights the formation of magnetite ore (Fe_3O_4) by reduction of hematite iron ore (Fe_2O_3) . Here, Fe and Fe_3O_4 are magnetic, while FeO and Fe_2O_3 are non-magnetic in nature.

2. MATERIALS AND METHODOLOGY EMPLOYED IN THE STUDY

2.1. Materials. A sample of low-grade hematite iron ore was obtained from the Dalli mines in Chhattisgarh, India. The collected sample was characterized by density determination, analysis of particle sizes, and wet chemical analysis performed to determine the metal content on a size-by-size basis, microscopic studies, XRD, FESEM, etc. Low ash content bituminous coal was used as a reductant in magnetizing roasting. Proximate analysis and determination of the calorific value were conducted to characterize the sample, particle size distribution, and specific gravity. The coal was crushed to

100% passing the 210 μm size for use as a reducing agent in the experiments.

2.2. Methodology. A large quantity of impurities, including silica, alumina, and phosphorus, is present in many naturally occurring iron ore deposits, which lower the iron concentration. By eliminating these impurities, beneficiation seeks to raise the iron content above 60% Fe, preparing the ore for iron and steel production; low-grade iron ore deposits could not be commercially viable for use in the direct production for steel industries. It is conceivable to extract and treat these resources on an economically sound basis by beneficiating the ore to increase its grade. Beneficiation techniques occasionally result in more energy-effective iron and steel production. For instance, employing higher-grade iron ore lowers the amount of energy used in the blast furnace, lowering greenhouse gas emissions. Figure 1 shows work

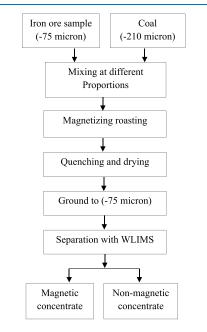


Figure 1. Experimental flowchart diagram for ore beneficiations.

flowchart for beneficiation process followed in the present investigation. The ore fineness was prepared below 75 μ m fineness and thoroughly mixed with coal fines in different proportions for experimental purposes.

For the magnetizing roasting purpose, iron-coal blending has been done in a silica crucible and was kept in the muffle furnace without placing a cover at different temperature levels for a designated time. The roasted product was quenched and then ground to 100% pass through $-75 \ \mu m$ in agate mortar. The roasted and ground samples underwent a wet lowintensity magnetic separation to confirm the magnetization process of hematite phases. An effective method for identifying and measuring the mineral phases found in powder ore (Iron ore) is X-ray diffraction (XRD) analysis. It offers important details on the ore's mineral makeup and crystalline structure.

In the mining and metallurgical sectors, XRD analysis offers crucial information about the mineralogical composition of iron ore samples, which is useful for ore characterization and process optimization. It can assist in determining the ore's appropriateness for particular uses and comprehending its behavior during processing. The grade of the magnetic concentrate was determined using wet chemical analysis. XRD and Microscopic studies were carried out in order to analyze the conversion of hematite into magnetite through phase transformation on roasted and magnetic separation concentrate. The complementary information they give on the mineralogical, textural, and structural characteristics of the sample are examined using optical microscopy and scanning electron microscopy (SEM), two significant methods used to examine iron ore powder. SEM provides high-resolution imaging, elemental analysis, and crystallographic information at smaller scales, whereas optical microscopy is great for macroscopic views and mineral identification. For the purpose of ore exploration, processing, and quality control in the mining and metallurgical sectors, the combination of these approaches enables a more thorough understanding of the mineralogy, texture, and microstructure of the ore.

3. CHARACTERIZATION STUDIES

The specific gravity of the received sample was measured using a pycnometer and was determined to be 3.85. The metal content in the ore sample was found to be 53.17% with main iron mineral phases as hematite (Fe₂O₃) and quartz (SiO₂) as shown in Table 1. The Fe (T) includes all the available Fe forms of hematite, magnetite, and goethite minerals. Microscopic examinations unveiled that the predominant iron mineral within the ore body is Fe₂O₃, constituting 65% of the composition, with significant proportions of martite and magnetite as well. The detailed findings are presented in Table 2.

Table 2. Mineralogical Composition of Iron Ore Fines

minerals	percentage (%)
hematite	65
goethite/limonite	10
clay + gibbsite	7
quartz + feldspar	10
martitized magnetite	2

The proximate analysis conducted on the bituminous coal sample employed in the study showed 11.56% ash, 4.03% moisture, 32.14% volatile matter, and 52.25% fixed carbon. The coal sample's calorific value, determined through bomb calorimeter analysis, was measured at 7039 J/kg. Figure 2 shows the distribution and interlocking pattern of iron and silicate minerals. The sample consists of hematite as predominant mineral and present in fine and medium sized grains. At a mesh size of -72, the majority of hematite grains exhibit independence, while the remaining ones are intertwined with goethite and silicate minerals.

Table 1. Iron Ore Fines Chemical Composition by Wet Chemical Analysis and XRF Analysis

	chemical compound								
fines of iron ore	Fe(T)	SiO ₂	Al_2O_3	LOI	CaO	MgO	TiO ₂	MnO	P_2O_5
weight percentage (%)	53.17	10.7	4.5	3.69	0.4	0.74	0.4	0.2	0.12

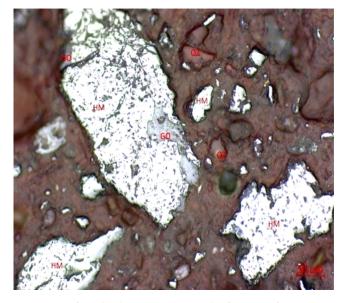


Figure 2. Reflected light micro photograph of iron ore fines.

Quartz and feldspar grains are predominantly fine to medium-sized. When sieved at -72 mesh size, the majority of quartz-feldspar grains are unbound, while the remains are intertwined with opaque materials. Little quartz-feldspar grains also contain opaque materials as inclusion. Clay is characterized by medium-sized grains with a significant portion of these grains being interlocked with iron and silicate minerals.

3.1. XRD, FE-SEM, & Mineralogical Studies. For XRD analysis of iron ore, fines with $-75 \ \mu$ m size was prepared and the analysis was carried out with the Cu K α radiation, at 30 mA current and 40 kV (Figure 3). Present studies revealed that the presence of mineral phases of hematite constitutes the primary iron-bearing mineral, with magnetite and goethite phases present in a minor proportion. Quartz was found to be as the major gangue mineral while gibbsite, feldspar, and pyrolusite as the minor amounts of gangue minerals (Figure 4).

3.2. Beneficiation Studies. The research aimed to explore how process parameters influence the magnetization process. The pertinent process parameters are given in Table 3. Magnetic separation was employed in the beneficiation process to track the iron concentrate's grade and recovery. Within the magnetizing roasting process, key factors affecting performance encompass the quality of iron ore, reduction temperature, roasting duration, and the coal percentage.

Coal to ore ratio 4.0 was fixed based on the stoichiometry equation between hematite and carbon and based on the literature; the reaction temperature of 700 °C was fixed for the theoretical iron-oxygen-carbon equilibrium curve, and 40 min of roasting time was used. The roasted samples were ground and subjected to beneficiation by using magnetic separation. Preliminary magnetic separation experiments were conducted on raw iron ore fines at varying magnetic field levels (800, 1200, 1600, and 2000 gauss). In order to assess the appropriate roasting processes, parameters on magnetic Fe content and recovery in magnetic fraction were studied with variable gauss levels. Before carrying out magnetizing roasting studies, wet low intensity magnetic separation (WLIMS) was carried out on raw ore fine samples to fix the optimum range of magnetic intensity value for this given hematite ore.

Samples, processed to have a particle size distribution of $-75 \ \mu$ m, were subjected to experimentation via wet low-intensity magnetic separation utilizing a Davis tube tester. Magnetic intensity levels were systematically varied across 800, 1200, 1600, and 2000. The outcomes of the initial investigations are depicted in Figure 5. It was found that 1600 gauss is the optimum magnetic intensity with grade and recovery values of 53.35 and 53.84%, respectively.

3.3. Effect of Feed to Coal Ratio on Magnetizing Roasting. In order to optimize the content of coal, different proportionate coal—iron ore fines were placed for experimental run. The amounts of coal in the iron—coal mix were kept at 3.6, 4.0, 4.4, and 4.8%. The experiments were conducted with a consistent temperature set at 700 °C, and the duration of each experiment was fixed at 30 min. The magnetic field intensity of 1600 gauss was maintained during magnetic separation. The

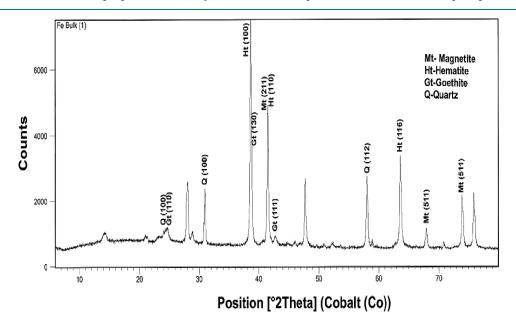


Figure 3. Hematite iron ore XRD analysis.

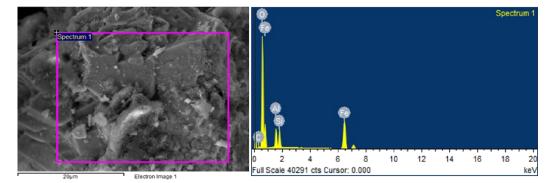


Figure 4. SEM image and EDS analysis of iron ore.

Table 3. Parameters	Used	in th	e Ex	periment
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experimental parameter	level 1	level 2	level 3	level 4
reductant, %	3.6	4	4.4	4.8
temperature, °C	650	700	750	800
time, min	15	30	45	60

highest grade of magnetic concentrate was achieved with a coal content of 4.4%; it has been increased to 59.8% Fe from the 53.17% of feed grade (Figure 6); and the recovery was reported as 61.35%. With an increase in the coal percent to 4.8%, the increase in the magnetic content was observed to be sluggish with a marginal increase in the recovery. This is due to the increasing coal percent evaluating more CO gas, which leads to the oxidation of converted magnetite.

3.4. Effects of Roasting Temperature. Roasting temperature is considered to be the most influencing factor for chemical changes that occur in iron ore fines. The experiments were conducted at temperatures of 650, 700, 750, and 800 °C. The studies were carried out at 4.4% coal content and for a fixed duration of 30 min in all the cases (Figure 7).

As shown in Figure 7, it can be observed that as the roasting temperature increases, the grade of the magnetic concentrate increases, reaching its peak at 62.35% Fe at 800 °C. Moreover, the recovery also exhibits an upward trend with higher temperatures. It reported a maximum recovery of 68.65% at a temperature of 800 °C, at which the grade was also found to be high.

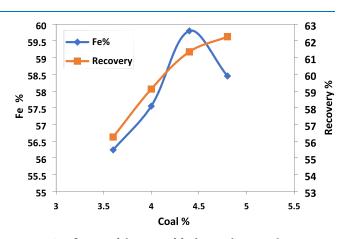


Figure 6. Influence of the ratio of feed to coal on metal recovery at temperature 700 $^\circ C$ and duration of 30 min.

3.5. Effect of Roasting Time. In this experimental run, iron ore fine samples with optimized 4.4% coal content were placed at the fixed optimized temperature of 800 °C with varying roasting time. The roasting duration was maintained at intervals of 15, 30, 45, and 60 min, and the experimental outcomes are presented in Figure 8.

The findings demonstrated that an extension in the roasting duration led to enhancements in both the grade and recovery of valuable components. At 15 min of roasting time, the grade was found to be 55.85% with a recovery of 55.65%. With increasing time, a linear enhancement in magnetic concentrate

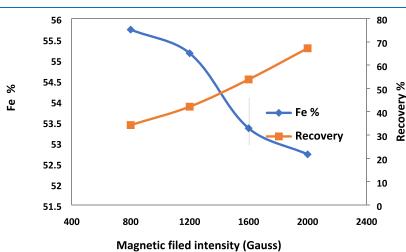


Figure 5. Effect of Gauss levels on grade and recovery.

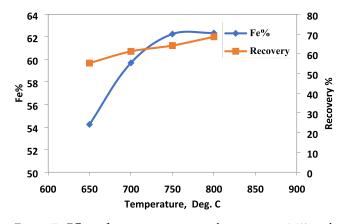


Figure 7. Effect of temperature on metal recovery at 4.4% coal, duration of 30 min.

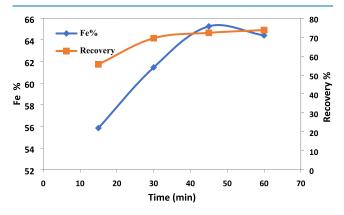


Figure 8. Effect of time on metal recovery at 4.4% coal and temperature of 800 $^\circ \rm C$

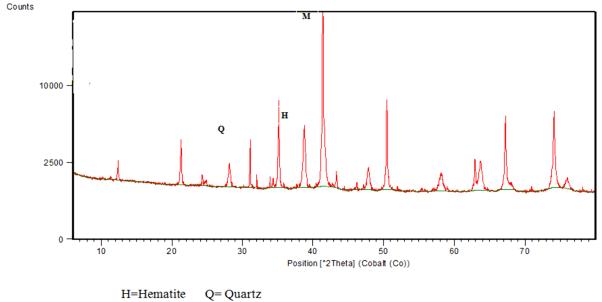
grade as well as recovery were observed. Nonetheless, when the duration exceeded 30 min, the positive impact on both grade and recovery became more pronounced. Extending the roasting duration after 45 min, it has been reported that there is a starting of declined recovery downfall.

3.6. Characterization Studies of Roasted Samples. To confirm the influence of pretreatment (roasting), X-ray diffraction (XRD) analysis and optical microscopic examination of the samples were conducted after the roasting process. The characterization of the product after magnetic separation was also carried out.

Figure 9 displays the XRD pattern of the roasted sample, which underwent roasting with a coal content of 4.4%, temperature of 800 °C, and time of 45 min; meanwhile, compared with analysis of the XRD pattern of the initial ore, it becomes evident that the transformation of the hematite phase into the magnetite phase occurred, with optimal results achieved by maintaining the coal-to-ore ratio under optimum conditions of temperature and time. Also, it has been found that even though transformation to the magnetic phase was taking place in considerable amounts, other small amounts of hematite peaks were defined in XRD, and quartz peaks were also evaluated in Figure 9, showing the XRD pattern of the roasted sample.

Figure 10 shows the XRD pattern for the magnetic concentrated roasted sample; magnetic concentration was carried out at a fixed magnetic field intensity of 1600 gauss as it has been optimized testing on raw hematite fine ore at different magnetic field intensities. Each magnetized product was subjected to magnetic separation at the 1600 gauss intensity. The XRD pattern of magnetic concentrate revealed that an optimum recovery of 71% and grade of 56.15 Fe % were reported at 4.4 coal %, temperature of 800 °C, and time of 45 min.

Figure 11a,b shows the optical micro images of the magnetized roasted product and magnetized magnetic concentrate product. In Figure 11a, a micrometer-sized magnetite is dispersed with the presence of silica matrix and the magnetic concentrated product image reveals the appearance of the maximized magnetite product.



M=Magnetite

Figure 9. Roasted sample at 4.4 coal%, temperature of 800 °C, and time of 45 min.

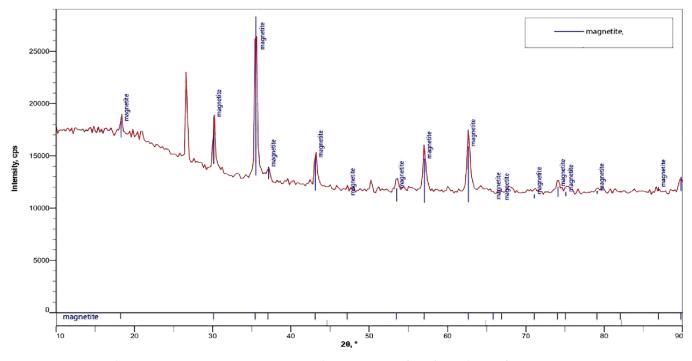


Figure 10. Magnetized magnetic separation concentrate at 4.4 coal%, temperature of 800 °C, and time of 45 min.

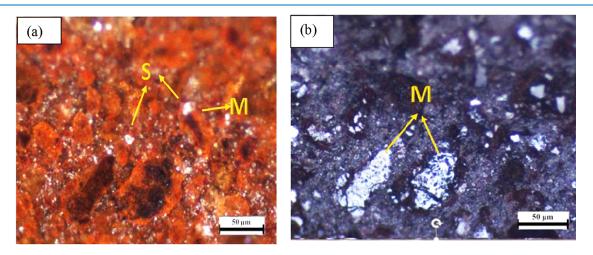


Figure 11. Optical microscopy study of ore (a) after roasting and (b) after magnetic separation.

It is clearly visible from the study (Figures 9 and 11a) that magnetizing roasting exerts a substantial influence on the characteristics of the hematite iron ore specimen. The presence of higher peaks of magnetite in the roasted samples (Figure 9) in comparison with the feed samples (Figure 3) clearly indicates that the magnetizing roasting might be a viable approach for the preliminary treatment in the processing of low-grade hematite iron ore fines. The postmagnetic separation product was subjected to characterization through X-ray diffraction (XRD) and image analysis, as depicted in Figures 10 and 11b, respectively. The overall process confirms the conversion of hematite into magnetite and enrichment of magnetite in the concentrate after magnetic separation.

4. CONCLUSIONS

Magnetizating roasting exhibited a pronounced impact on the magnetic attributes of the hematite ore, resulting in the transformation of hematite into magnetite through selective magnetizing roasting. The optimum results were obtained at the experimental conditions of 800 °C temperature, 45 min roasting time, and 4.4% coal content with 100% passing 210 μ m mixed with the iron ore. Upon magnetic separation, the best result with 65.25% Fe was obtained with a recovery value of 72.3% in the concentrate. The reported product had significant improvement in grade, and the partially reduced ore may be possibly used to improve the blast furnace productivity rate. The other process variables at optimum conditions were the 1600 gauss magnetic field and particle fineness of 100% passing 75 micrometers. This study confirmed the significant impact of temperature, reductant percentage, and roasting time on the performance of magnetization and hence the magnetic separation process.

AUTHOR INFORMATION

Corresponding Author

Shravan Kumar – Department of Fuel Minerals & Metallurgical Engineering, Indian Institute of Technology (ISM) Dhanbad, Dhanbad, Jharkhand PIN - 826 004, India; Email: shravan@iitism.ac.in

Authors

Prabhu Chand Kukkala – Department of Fuel Minerals & Metallurgical Engineering, Indian Institute of Technology (ISM) Dhanbad, Dhanbad, Jharkhand PIN - 826 004, India

Akhileshwar Nirala – Department of Fuel Minerals & Metallurgical Engineering, Indian Institute of Technology (ISM) Dhanbad, Dhanbad, Jharkhand PIN - 826 004, India; Department of Mechanical Engineering, Galgotias College of Engineering and Technology, Greater Noida 201310, India

Mohammad Amir Khan – Department of Civil Engineering, Galgotias College of Engineering & Technology, Greater Noida 201310, India; @ orcid.org/0009-0006-3612-8948

Meshel Q. Alkahtani – Civil Engineering Department, College of Engineering, King Khalid University, Abha 61421, Saudi Arabia

Saiful Islam – Civil Engineering Department, College of Engineering, King Khalid University, Abha 61421, Saudi Arabia

Complete contact information is available at: https://pubs.acs.org/10.1021/acsomega.3c06802

Notes

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